# SYNOPSIS

## Synthesis, Characterization and Applications of Gemini Surfactants

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# Introduction

Surfactants are compounds which lower the surface tension between two liquids or between a liquid and a solid. They may act as detergents, wetting agents, emulsifiers, and foaming agents. Chemically, surfactants are amphiphilic organic compounds, which mean they contain hydrophobic groups (their tails) and hydrophilic groups (their heads).The tails of most surfactants are fairly similar consisting of a hydrocarbon chain which can be branched, linear and aromatic. Thus, surfactant consists of both, a water insoluble component and a water soluble component. Due to this property surfactants can diffuse in water and absorb at interfaces between air and water. Important property of amphiphilic surfactant molecules is their ability to aggregate in solution. The aggregation process depends on the nature of amphiphilic species and solvent in which they are dissolved. The aggregation of surfactant molecules results into the formation of micelle at a definite concentration of surfactant in solution. The process of formation of micelle is called micellization and the definite concentration of surfactant molecules in solution is called critical micelle concentration (CMC).

Surfactants show interesting interfacial and bulk properties and have a wide variety of uses which are mostly met by conventional representatives [1]. Changes in molecular structure and type to improve their properties have attracted the attention of many researchers [2, 3]. This results into preparation of new generation surfactants like Gemini surfactants. To provide greater surface activity, scientists are in search of newer surfactants and the preparation started with bis-surfactants, which are later renamed as Gemini surfactants.

Gemini surfactant is the family of surfactant molecules possessing more than one hydrophobic tail and hydrophilic head group. They are different from conventional surfactants, as a Gemini has in sequence a hydrocarbon chain, an ionic group, a spacer, a second ionic group and another hydrocarbon tail. Gemini surfactants are found to be more surface active than conventional surfactants [4]. A great deal of variation exists in nature of species depending on nature of the spacers or conventional surfactants used. For example, short as well as long methylene groups, polar and non-polar groups may be used as spacers. On the other hand, the ionic groups can be positive or negative whereas the non-ionic groups can be polyether or sugar. The great majority of Gemini surfactants have symmetrical structures with two identical polar groups and two identical chains but some unsymmetrical Geminis and Geminis with three or more polar groups or tails have recently been reported [5].

Many studies have also been reported on applications of Gemini surfactants in different fields. For instance, the antimicrobial activity and corrosion efficiency against hydrochloric acid medium have been investigated for some selected surfactants [6]. Gemini surfactants against brass in 3N $HNO_3$ solution have shown excellent corrosion inhibition properties [7]. The weight loss method shows that inhibition efficiency of Gemini surfactants increases with the increase in temperature and concentration against 1M HCL solution for mild steel at temperature range (303.15-333.15) K [8]. The electrochemical effect of cationic Gemini surfactants on corrosion inhibition properties shows that, the inhibition efficiency increases with surfactant concentration [9]. The anti-corrosive activities of some novel surfactants based on vegetable oils have also been studied and are found good corrosion inhibitor [10]. The use of modified Gemini surfactants has been reported in order to improve physio-chemical and biological behaviour of drugs like melpharan-$\beta$-cyclo dextrin [11]. These surfactants are good choice for solubilizing hydrophobic materials such as Amyloid -$\beta$-peptides which cause Alzheimer disease [12]. Novel glycerol and cotton seed oil based non-ionic Gemini surfactants have been reported to possess good solubilizing and wetting behaviour [13].

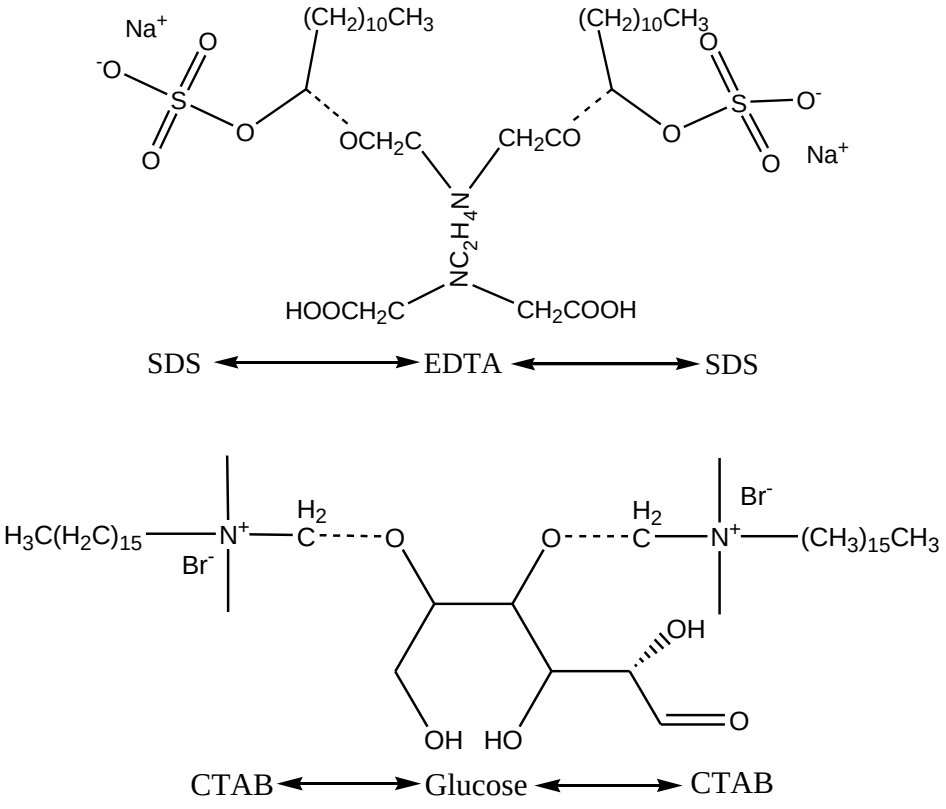


**Fig. 1** Structure of Gemini surfactants of SDS and CTAB

Therefore, in this study, we intend to synthesize Gemini surfactants of SDS and CTAB (Figure 1) by chemical refluxing method with two precursors, glucose and bi-sodium salt of ethylene diamine tetra-acetic acid. As synthesized Gemini surfactants will be characterized by using NMR, FTIR, and surface tension measurement techniques, and will also be investigated for their anti corrosion activity.

## Literature Review

In late $17^{th}$ century while travelling on a ship, Banjamin Franklin observed that water does not get mixed with air. This provided a basic framework for properties of surfactants. In 1929 Kurt Von Neergaard performed experiments suggesting the presence of pulmonary surfactants and its relevance to the newborn's first breath. Researcher, John Clements identified surfactants and their role in the 1950s. Dr. Mary Auery soon after showed that the lungs of premature infants could not produce surfactants. To know more about surfactants more research was done in this field leading to synthesis of Gemini surfactants as discussed in following paragraphs.

Hait et al. [1] discovered a new class of surface active agents with two hydrophobic and hydrophilic groups. They investigated antibacterial properties of synthesized Gemini surfactants and found that these properties depend on length and type of spacer used.

Fatma et al. [2] synthesized anionic Gemini surfactants and studied their antimicrobial effect in simple as well as when they get mixed with another conventional non-ionic surfactant. They also studied micellization behaviour of these anionic Gemini surfactants.

Menger et al. [3] synthesized and observed the properties of Gemini surfactants. It was observed that Gemini surfactants were unable to aggregate without exposure of hydrocarbon tails in water. Surface tension studies of these Gemini surfactants shows that surfactants with shorter chain length had much lower critical micelle concentration as compared to surfactants with larger chain lengths.

Holmberg [4] prepared surfactants using amphiphiles produced by yeast or bacteria, amphiphiles containing natural hydrophobic tail and amphiphiles containing natural polar head groups by fermentation process. They also studied physico-chemical properties for surfactants prepared by organic synthesis.

Kallio et al. [5] studied the effect of a surfactant assisted synthesis on the electrochemical performance of a lithium iron phosphate ($LiFePO_4$) – Carbon nano tube composite electrode.

Sadek et al. [6] prepared some selective Gemini surfactants and studied about surface properties, biological activity and corrosion efficiency against hydrochloric acid medium. They also found that these surfactants are attractive for catalysis, adsorption applications, enhanced oil recovery and as a paint additive.

Sharma et al. [7] investigated corrosion inhibition of Gemini surfactants. The purpose of their research was to study corrosion inhibition of brass in 3N $HNO_3$ by Gemini surfactants.

Mobin et al. [8] discovered the corrosion inhibition characteristics of synthesized cationic Gemini surfactant. It was studied for mild steel in 1M HCl solution over a temperature range (303-333) K. They discovered that corrosion inhibition property of surfactant increases with their concentration.

Mohmoodi et al. [9] studied electrochemical effect of cationic Gemini surfactants and halide salts as corrosion inhibitors on carbon steel in acidic medium. They concluded that the inhibition efficiency increases with increase in surfactant concentration.

Michel et al. [10] synthesized beta-cyclo dextrin modified Gemini surfactant and developed it to incorporate Melpharan (drug type) in order to improve its physiochemical and biological behaviour.

Abbasov et al. [11] studied anti corrosive activities of some novel surfactants which were based on vegetable oils. It was found that tested compounds showed good corrosion inhibition at steel even at low concentration and this was related to the compact structure of these compounds.

Bahmani et al. [12] studied effect of Gemini surfactants on Amyloid Beta Aggregation. According to authors, properties of Gemini surfactants make them a good choice for stabilizing materials of high hydrophobicity such as Amyloid Beta peptides which cause Alzheimer disease.

Gawali et al. [13] prepared novel glycerol based non ionic Gemini surfactants. The effect of surfactant on solubilization of polar and non-polar solute was studied. It was concluded that new glycerol based non-ionic Gemini surfactants exhibit generally good solubilizing and wetting behaviour.

Yuan et al. [14] studied characteristics and harmful effects of surfactants on nature and concluded that branched surfactants are more harmful to nature as compared to straight chain surfactants.

Gagneton et al. [15] synthesised N-Acetylated Gemini surfactant and studied its surface active properties, antifungal activity which basically depends on chain length and spacer used for synthesis of these Gemini surfactants.

Wang et al. [16] studied the surface properties, aggregation behaviour and micellization thermodynamics of a class of Gemini surfactants with ethyl ammonium head groups. They also reported application of these synthesized compounds in future.

Gujral et al. [17] prepared asymmetrical Gemini surfactants and investigated their physical properties like surface tension, electrical conductivity, and critical micelle concentration.

Singh et al. [18] investigated mixed surfactant systems of lauryl alcohol based bisulfosuccinate anionic Gemini surfactants with conventional surfactant. They investigated the association and aggregation properties of these surfactants with a flexible methylene spacer in aqueous solution.

Han et al. [19] studied effects of the hydrocarbon chains and spacer groups of Gemini surfactants, salt, temperature and polymer on interfacial tension of crude oil water mixture. According to authors, length of alkyl chain and spacer group both affect properties of Gemini surfactants.

Chen et al. [20] studied monolayer effect of a Gemini surfactant with a rigid biphenyl spacer on its self crystallization at the air/liquid interface. X-ray crystallography revealed that a Gemini surfactant with biphenyl spacer can spontaneously be crystallized in air/liquid interface.

Patel et al. [21] prepared some zwitter ionic Gemini surfactants. Gemini surfactants with two methylene spacer from the EDTA and alkyl bromides have been synthesised and characterized from data obtained by NMR spectroscopic analysis.

Groth et al. [22] studied kinetics of self-assembly of Gemini surfactants and studied the kinetics of exchange, size and shapes in micellar systems of cationic surfactant dimers by using stopped flow technique.

Nagarajan et al. [23] reported a comparative analysis of free energies of Gemini surfactants which showed that short length of spacer play a central role in determining the equilibrium shape of aggregate.

Mathias et al. [24] investigated the aggregation number of Gemini surfactants with dimethylenephenylene spacer groups using fluorescence method and also found that long chain Geminis also have a critical micelle concentration greater than expected on the basis of plots of log CMC vs chain length for the shorter chain homologues.

Kozak et al. [25] has synthesized Gemini surfactant based on Bis –Imidazoliumalkoxy derivatives. They concluded that these Gemini surfactants are effective agents for delivery of nucleic acid.

John et al. [26] discussed about recent development in materials synthesis in surfactant systems and studied different structures which were formed when surfactant molecules gets self-assembled by intermolecular forces.

Joshi et al. [27] prepared Gemini surfactants using heterogeneous basic catalyst. In this tri ethanolamine and a fatty acid using MgO-$ZrO_2$ selectively gave the corresponding 2-(bis(2-hydroxyethyl)amino)ethyl alkanoate in good yield.

Asadov et al. [28] prepared surfactants based on palmitic acid and nitrogenous bases. They studied their properties including critical micelle concentration (CMC), surface excess etc. The results showed that some of synthesized complexes could be used as petro-collecting and dispersing agents for removing petroleum films from water surface.

Adawy et al. [29] prepared amide based cationic surfactant complexes with Cobalt and Copper and studied antimicrobial activity of these prepared surfactants via the inhibition zone diameter against different microorganisms. The results indicated that these metallocationic surfactants have good surface properties and good biological activity on broad spectrum of microorganisms.

Kumar et al. [30] prepared anionic Gemini surfactant using EDTA anhydride as spacer and named it as G16 Gemini surfactant. They found that, the synthesized surfactants are efficient agents to remove hardness of water as EDTA traps unwanted ions from water.

Kumar et al. [31] examined aqueous micellar solutions of bile salts, sodium cholate (NaC) and sodium deoxycholate (NaDC) by surface tension and analysed their adsorption behaviour by calculating interfacial parameters like maximum surface excess concentration ($\Gamma_{\max}$), minimum area per surfactant molecule ($A_{\min}$), surface pressure at CMC ($\Pi_{CMC}$), efficiency in reducing surface tension ($pC_{20}$), and adsorption at the air/water interface relation to micellization ($CMC/C_{20}$).

Chauhan et al. [32] investigated the interactions of the anionic surfactant sodiumdodecylsulfate (SDS) with aqueous polyethylene glycol (PEG), polyvinyl pyrrolidone (PVP) at different temperatures by using conductivity, density and speed of sound techniques. The nature of the process of micellization had evidenced from the magnitude of $\Delta G_m^o, \Delta H_m^o$ and $\Delta S_m^o$ values.

Chauhan et al. [33] studied effect of co-solvent N-methylacetamide on the micellization behaviour of anionic surfactant sodium dodecylsulphate (SDS) and cationic surfactant cetyltrimethylammonium bromide (CTAB) in aqueous solution by conductivity measurements.

Critical micelle concentration (CMC) values were also determined for SDS and CTAB in aqueous solutions of NMA from the conductivity versus surfactant concentration plots.

## Research Gap Identification

Although a number of research groups from all over the world synthesized Gemini surfactants and studied their properties like corrosion inhibition, antibacterial action, foaming agent and wetting properties, but there are many Gemini surfactants which are yet to discover. Moreover, the micellization behaviour of most of the Gemini surfactants yet has not been analysed in terms of interfacial parameters like surface excess at air/water interface ($\Gamma_{max}$), minimum area per surfactant molecule ($A_{min}$), surface pressure at the $CMC$ ($\Pi_{CMC}$), standard Gibb's free energy of adsorption ($\Delta G_{ad}^{o}$), standard free energy of micellization ($\Delta G_{m}^{o}$) and standard free energy of transfer ($\Delta G_{tr}^{o}$) to gain more clear insight into the surface effectiveness of Gemini surfactants in aqueous medium. Thus, sodium dodecyl sulphate (SDS) and cetyltrimethylammonium bromide (CTAB) are selected to synthesize Gemini surfactants and to investigate their micellization behaviour as well as corrosion inhibition efficiency.

## Objectives

1. To synthesize Gemini surfactant of sodium dodecyl sulphate (SDS) and cetyltrimethylammonium bromide (CTAB) using glucose and disodium salt of EDTA as spacers.
2. To characterize the synthesized Gemini surfactants by employing FTIR and NMR spectroscopic techniques.
3. To investigate the micellization behaviour of Gemini surfactants in aqueous medium by using surface tension measurements.
4. To use the synthesized Gemini surfactants as corrosion inhibitors.

## Proposed Methodology

### Synthesis of Gemini Surfactants

A solution of absolute ethanol containing bi-sodium salt of EDTA/Glucose (1 mmmol·kg$^{-1}$) will be taken in a round bottom flask in which SDS/CTAB (2.2 mmol·kg$^{-1}$) will be added [6].

Then 0.2 gm of NaOH will be added to the reaction mixture as the catalyst followed by acidification using aqueous solution of HCl. The reaction will then be refluxed for 8 hours at 323.15 K. Crystals of Gemini surfactants shall be purified and recrystallized in the ethanol before further study.

**Characterization of Gemini Surfactants**

Synthesized surfactants will be characterized for purity and molecular structures with the help of FTIR and $^1$H-NMR spectroscopic techniques by following standard procedure. Infrared spectra will be obtained on 600 FT-IR Agilent Technologies Instrument in the 4,000–400 $cm^{-1}$ range using KBr pellets.$^1$H-NMR spectra will be recorded on a JEOL 500 MHZ instrument with $CDCl_3$ solvent.

**Surface Tension Measurements**

Surface tension measurements will has been used to calculate the critical micelle concentration (CMC) values and other thermodynamic parameters like $\Gamma_{max}$, $A_{min}$, $\Delta G^o_{ad}$, etc. of synthesized Gemini surfactants. $\Gamma_{max}$, which represents a measurement of the effectiveness of adsorption of the surfactant, will be calculated using the Gibbs adsorption equation [34, 35].

$$\Gamma_{max} = -(1/2.303nRT)(\partial\gamma/\partial \log C)_{T,P} \quad (1)$$

where 'n' is the number of particles per molecule of the surfactant whose surface concentration varies with change in bulk concentration of the surfactant, and will be taken as '2' for the reason described earlier [35,36]. $(\partial\gamma/\partial \log C)_{T,P}$ is the slope of the $\gamma$ vs. log[Gemini surfactant] plots and has been determined by using linear fit to the $\gamma$ vs. log[Gemini surfactant]values in pre-micellar region [37].

The $A_{min}$ at the air–water interface will be evaluated according to the following equation [34, 38];

$$A_{min} = 10^{18}/\Gamma_{max} N_o \quad (2)$$

where, $N_o$ is Avogadro's number. The $\Pi_{CMC}$ known as surface pressure at $CMC$, is calculated as [34]

$$\Pi_{CMC} = \gamma_o - \gamma_{CMC} \quad (3)$$

where, $\gamma_o$ and $\gamma_{CMC}$ are the surface tension of the solvent and of the micellar solution at $CMC$ , respectively. The standard Gibbs free energy of adsorption, $\Delta G_{ad}^o$ will be calculated from the equation (4) [31].

$$\Delta G_{ad}^o = \Delta G_m^o - N_A A_{\min} \Pi_{CMC} \quad (4)$$

where $\Delta G_m^o$ known as standard free energy of micellization of Gemini surfactants is determined using equation (5).

$$\Delta G_m^o = RT \ln(X_{CMC}) \quad (5)$$

The $CMC$ and $\Gamma_{\max}$ values will be further used to estimate the standard free energy of transfer, $\Delta G_{tr}^o$ of Gemini surfactants from the bulk to the micellar region using the equation (6).

$$\Delta G_{tr}^o = -RT \ln[100\Gamma_{\max} N_A^{1/3} / (CMC)^{2/3}] \quad (6)$$

**Corrosion Inhibition Properties**

The corrosion inhibition effect of Gemini surfactants on 'Tin' in 2 mol·kg$^{-1}$ HCl solution will be studied by simple weight loss method [5]. In this method two stripes of tin of equal weights, one carrying a layer of Gemini surfactant and other without Gemini surfactant will be dipped in 2 mol·kg$^{-1}$ HCl solution for 20 hours at room temperature. After that, the layers of corrosion on strips will be removed and weight of stripes will be measured.

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